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BENZYL-BENZOATE IN CIGARETTE
SMOKE.

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ABSTRACT

Silicic acid chromatography of the nitromethane-soluble portion of the neutral fraction of smoke condensate from domestic cigarettes yielded a residue which showed absorption in the infrared region of the spectrum attributable to "ester" and NH or -OH. Gas chromatography of the residue on SE-30/glass beads indicated the presence of at least 20 components, with major peaks eluting at 140° and 185°C. Infrared spectral analysis of the later peak was indicative of an aromatic ester; additional data obtained by mass spectroscopy, hydrolysis, and retention time studies showed the peak to contain benzylbenzoate (400 µg/100 cigarettes). This ester has not been reported previously in cigarette smoke. The gas chromatographic peak eluting at 140°C. was identified as skatole on the basis of gas chromatographic and spectro-photometric characteristics. Preliminary examination of some of the other peaks indicate the presence of one or more aromatic esters, carbazole-like compounds, and an aromatic aldehyde.

REVIEW BY M. L. GILFOYLE

The residue from the nitromethane soluble portion of the neutral fraction of smoke condensate from 5000 domestic cigarettes was investigated. The infrared spectrum of the residue indicated an ester plus an -OH or NH band at 3500cm⁻¹.

The residue was gas chromatographed under the following conditions:

Column: 4' x $\frac{1}{8}$ " - 0.25% SE-30/Glass Beads

Column Temperature: 65°C - 300°C at 4°C/min.

Flow Rate: 60 ml/min. He

Detector Temperature: 315°C

Injection Port Temperature: 325°C

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The gas chromatogram showed twenty peaks, of which there were two major peaks. The peak eluted at 140°C was skatole and the peak eluted at 185°C was benzyl benzoate, which had not been identified in smoke prior to this time.

The infrared analysis of the eluant at 185°C indicated an aromatic ester. The mass spectrum showed a compound with molecular weight 212. Based on retention time studies, hydrolysis, mass spectrometric and infrared data, this ester was identified as benzyl benzoate.

The following is the elution pattern of nitromethane subfractions off alumina:

↓
Hydrocarbons
Esters, Ethers
Skatole and other Methyl Indoles
Indole
Carbazoles (methyl carbazoles)
↓
Resinous Materials

All fractions were gas chromatographed. The latter four fractions were run under the following conditions:

Column: 10' x $\frac{1}{8}$ " - 20% SE-30/Chromosorb W

Column Temperature: 240°C

Flow Rate: 60 ml/min. He

Detector: Flame ionization

Detector Temperature: 290°C

Injection Port Temperature: 260°C

<u>Retention Time</u>	<u>Components</u>
1 minute	carbazole
2½ minutes	skatole
3 minutes	indole
10 minutes	methyl carbazole
14 minutes	dimethyl carbazole

Composition of Nitromethane Subfraction

<u>Compound</u>	<u>Criteria for Identification</u>
Skatole	IR, UV, GC, Ehrlich Test
Methyl Indole	IR, MS
Dimethyl Indole	IR, MS
Trimethyl Indole	IR, MS
Indole	GC
Carbazole	IR, UV, GC, MS
Methyl Carbazole	IR, UV, GC, MS
Dimethyl Carbazole	IR, UV, GC, MS
Benzyl Benzoate	IR, UV, GC, MS, Hydrolysis
Benzyl Cinnamate	IR, MS
Aromatic Ether (known but not reported)	IR, MS

The author reported 4 μ g of benzyl benzoate per cigarette and a large amount of skatole, but this was not measured.